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Integrated Data Collection Analysis (IDCA) Program - Ammonium Nitrate

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ABSTRACT

The Integrated Data Collection Analysis (IDCA) program is conducting a proficiency study for Small-Scale Safety and Thermal (SSST) testing of homemade explosives (HMEs). Described here are the results for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis of ammonium nitrate (AN). AN was tested, in most cases, as both received from manufacturer and dried/sieved. The participants found the AN to be: 1) insensitive in Type 12A impact testing (although with a wide range of values), 2) completely insensitive in BAM friction testing, 3) less sensitive than the RDX standard in ABL friction testing, 4) less sensitive than RDX in ABL ESD testing, and 5) less sensitive than RDX and PETN in DSC thermal analyses.

This effort, funded by the Department of Homeland Security (DHS), is putting the issues of safe handling of these materials in perspective with standard military explosives. The study is adding SSST testing results for a broad suite of different HMEs to the literature. Ultimately the study has the potential to suggest new guidelines and methods and possibly establish the SSST testing accuracies needed when developing safe handling practices for HMEs. Each participating testing laboratory uses identical test materials and preparation methods wherever possible. Note, however, the test procedures differ among the laboratories. The testing performers involved are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (NSWC IHD), Sandia National Laboratories (SNL), and Air Force Research Laboratory (AFRL/RXQL). These tests are conducted as a proficiency study in order to establish some consistency in test protocols, procedures, and experiments and to compare results when these testing variables cannot be made consistent.

Keywords: Small-scale safety testing, proficiency test, impact-, friction-, spark discharge-, thermal testing, round-robin test, safety testing protocols, HME, RDX, potassium perchlorate, potassium chlorate, sodium chlorate, sugar, dodecane, PETN, carbon, ammonium nitrate.

Explosives Safety Testing of Homemade Explosives

Integrated Data Collection Analysis Program

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1 INTRODUCTION

The IDCA Proficiency Test was designed to assist the explosives community in comparing and perhaps standardizing inter-laboratory Small-Scale Safety and Thermal (SSST) testing for improvised explosive materials (homemade explosives or HMEs) and aligning these procedures with comparable testing for typical military explosives[1]. The materials for the Proficiency Test have been selected because their properties invoke challenging experimental issues. Many of these challenges are not normally encountered with military type explosives. To a large extent, the issues are centered on the physical forms and stability of the improvised materials.

Often, HMEs are formed by mixing oxidizer and fuel precursor materials, and typically, the mixture precursors are combined shortly before use. The challenges to produce a standardized inter-laboratory sample are primarily associated with mixing and sampling. For solid-solid mixtures, the challenges primarily revolve around adequately mixing two powders on a small scale, producing a mixture of uniform composition—particle size and dryness often being a factor—as well as taking a representative sample. For liquid-liquid mixtures, the challenges revolve around miscibility of the oxidizer with the fuel causing the possibility of multiphase liquid systems. For liquid-solid mixtures, the challenges revolve around the ability of the solid phase to mix completely with the liquid phase, as well as minimizing the formation of intractable or ill-defined slurry-type products.

Table 1. Materials for IDCA Proficiency study

Oxidizer/Explosive	Fuel	Description
Potassium perchlorate	Aluminum	Powder mixture
Potassium perchlorate	Charcoal	Powder mixture
Potassium perchlorate	Dodecane[1]	Wet powder
Potassium chlorate	Dodecane[1]	Wet powder
Potassium chlorate as received	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Potassium chlorate -100 mesh[3]	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Sodium chlorate	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Ammonium nitrate		Powder
Bullseye® smokeless powder[4]		Powder
Ammonium nitrate	Bullseye® smokeless powder[4]	Powder mixture
Urea nitrate	Aluminum	Powder mixture
Urea nitrate	Aluminum, sulfur	Powder mixture
Hydrogen peroxide 70%	Cumin	Viscous paste
Hydrogen peroxide 90%	Nitromethane	Miscible liquid
Hydrogen peroxide 70%	Flour (chapatti)	Sticky paste
Hydrogen peroxide 70%	Glycerine	Miscible liquid
HMX Grade B		Powder
RDX Class 5 Type II		Powder (standard)
PETN Class 4		Powder (standard)

1. Simulates diesel fuel; 2. Contains 3 wt. % cornstarch; 3. Separated to pass 100 mesh; 4. Alliant Bullseye® smokeless pistol gunpowder.

The IDCA has chosen several formulations to test that present these challenges. Table 1 shows the materials selected for the Proficiency Test and the Description column describes the form of the resulting mixture. Details of the results from the Proficiency Test for the materials examined are documented in IDCA reports—RDX first testing[2], RDX second testing[3], RDX testing comparison[4], $KClO_3$/sugar (separated with a 100-mesh sieve)[5], $KClO_3$/sugar (as received)[6], $KClO_3$/Dodecane[7], $KClO_4$/Dodecane[8], $KClO_4$/Al[9], $KClO_4$/Carbon[10], $NaClO_3$/sugar[11], PETN[12] and Methods[13].

Evaluation of the results of SSST testing of unknown materials, such as the HMEs in Table 1, is generally done as a relative process, where an understood standard is tested alongside the HME. In many cases, the standard employed is PETN or RDX. The standard is obtained in a high purity, narrow particle size range, and measured frequently. The performance of the standard is well documented on the same equipment (at the testing laboratory), and is used as the benchmark. The sensitivity to external stimuli and reactivity of the HME (or any energetic material) are then evaluated relative to the standard.

Most of the results from SSST testing of HMEs are not analyzed any further than this. The results are then considered in-house. This approach has worked very well for military explosives and has been a validated method for developing safe handling practices. However, there has never been a validation of this method for HMEs. Although it is generally recognized that these SSST practices are acceptable for HME testing, it must always be kept in mind that HMEs have different compositional qualities and reactivities than conventional military explosives.

The IDCA is evaluating SSST testing methods as applied to HMEs. In addition, the IDCA is attempting to understand, at least in part, the laboratory-to-laboratory variation that is expected when examining HMEs. The IDCA team has taken several steps to make this inter-laboratory data comparison easier to analyze. Each participating laboratory uses materials from the same batches and follows the same procedures for synthesis, formulation, and preparation. In addition, although the Proficiency Test allows for laboratory-to-laboratory testing differences, efforts have been made to align the SSST testing equipment configurations and procedures to be as similar as possible, without significantly compromising the standard conditions under which each laboratory routinely conducts their testing.

The first and basic step in the Proficiency Test is to have representative data on a standard material to allow for basic performance comparisons. Table 1 includes some standard military materials. Class 5 Type II RDX was chosen as the primary standard, and Class 4 PETN was chosen as a secondary material. These materials have been tested in triplicate and RDX was tested throughout the IDCA Proficiency Test.

The subject of this report, AN, is the third single component material examined in the Proficiency Test, and the first in the AN-Gunpowder mixture series. Pure Gunpowder and the AN-Gunpowder mixture will be analyzed and compared in the future. The testing performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center (NSWC IHD), and Air Force Research Laboratory (AFRL), Tyndall AFB.

2 EXPERIMENTAL

General information. All samples were prepared according to IDCA methods on drying and mixing procedures[14,15]. Briefly, the sample was dried in an oven at 60°C for 16 h, then cooled and stored in a desiccator until use. The AN was Fisher Brand, Catalog Number A676, Lot #086459. For characterization, the AN was dried and separated through a 100-mesh sieve. The average particle properties were measured by laser diffraction light scattering method using Microtracs Model FRA9200. The TGA data was collected on a TA600 DSC with a Pfeiffer Evolved Gas Analyzer attached on the gas outlet.

Testing conditions. Table 2 summarizes the SSST testing conditions used by the laboratories that participated in the analyses of the AN. SSST testing data for the individual participants was obtained from the following reports: Small Scale Safety Test Report for Ammonium Nitrate (LLNL)[16], 50188 N Ammonium Nitrate (LANL)[17], and 50188 N-2 AN Dried and as Received (LANL)[18], AN Report (100 Mesh Dried at 60°C) (IHD)[19], Effect of Pan Type on Decomposition of Ammonium Nitrate (IHD)[20], DSC Characterization of Ammonium Nitrate Dried at 60°C (IHD)[21], Ammonium Nitrate (AN) dried at 60°C (AFRL)[22], and DSC Analysis Report—Ammonium Nitrate (RR) before Heating (AFRL)[23].

Table 2. Summary of conditions for the analysis of RDX (All = LANL, LLNL, IHD, AFRL)

Impact Testing
1. Sample size—LLNL, IHD, AFRL 35 ± 2 mg; LANL 35 or 40 ± 2 mg
2. Preparation of samples—All, dried and sieved per IDCA drying methods[14]; LANL as received
3. Sample form—All, loose powder
4. Powder sample configuration—All, conical pile
5. Apparatus—LANL, LLNL, IHD, Type 12; AFRL, MBOM with Type 12 tooling*
6. Sandpaper—All (180-grit garnet dry); LLNL (120-grit Si/Carbide wet)
7. Sandpaper size—LLNL, IHD, AFRL, 1 inch square; LANL, 1.25 inch diameter disk dimpled;
8. Drop hammer weight—All, 2.5 kg
9. Striker weight—LLNL, IHD, AFRL, 2.5 kg; LANL 0.8 kg
10. Positive detection—LANL, LLNL, microphones with electronic interpretation as well as observation; IHD, AFRL, observation
11. Data analysis—All, modified Bruceton; LANL and AFRL, Neyer also

Friction analysis
1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA procedures[14]
3. Sample form—All, powder
4. Sample configuration—All, small circle form
5. Apparatus—LANL, LLNL, IHD, BAM; IHD, AFRL, ABL
6. Positive detection—All, by observation
7. Room Lights—LANL and AFRL on; and LLNL off; IHD, BAM on, ABL off
8. Data analysis—LLNL modified Bruceton (log-scale spacing) and TIL; LANL and IHD, modified Bruceton (linear spacing) and TIL; AFRL, TIL

ESD
1. Sample size—All ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA drying methods[14]
3. Sample form—All, powder
4. Tape cover—LANL, scotch tape; LLNL, Mylar; IHD and AFRL, none
5. Sample configuration—All, cover the bottom of sample holder
6. Apparatus—LANL, IHD, AFRL, ABL; LLNL, custom built*
7. Positive detection—All, observation; LLNL IR gas (CO_2/CO)
8. Data analysis methods—All, TIL

Differential Scanning Calorimetry
1. Sample size—All ~ <1 mg
2. Preparation of samples—All, dried and sieved per IDCA procedures[14], and as received
3. Sample holder—All, pinhole; LLNL, TA sealed; IHD SWISSI high pressure
4. Scan rate—All, 10°C/min
5. Range—All, 40 to 400°C
6. Sample holder hole size—LANL, IHD, AFRL, 75 μm; LLNL 50 μm
7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920 and Setaram Sensys; IHD, AFRL, TA Instruments Q1000*

Footnotes: *Test apparatus, *Impact:* LANL, LLNL, IHD—ERL Type 12 Drop Weight Sensitivity Apparatus, AFRL, SNL— MBOM modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD, SNL—BAM Friction Apparatus, LANL, IHD, AFRL—ABL Friction Apparatus; *Spark:* LLNL, LANL, IHD, AFRL, SNL—ABL Electrostatic Discharge Apparatus, LLNL—custom-built Electrostatic Discharge Apparatus; *Differential Scanning Calorimetry:* LANL—TA Instruments Q1000, Q2000, LLNL—TA Instruments 2910, 2920, Setaram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

3 RESULTS

3.1 Ammonium Nitrate

In this Proficiency Test, all testing participants are required to use materials from the same batch, and mixtures are to be prepared by the same methods. However, the actual testing procedures can be different. These differences are described in the IDCA report on method comparisons[13], which compares procedures by each testing category. LANL, LLNL, IHD, and AFRL participated in this testing.

Table 3. Certificate of analysis (COA) for ammonium nitrate use in the Proficiency Test

Catalog Number	A676	Mfg. Date	2/4/2009
Lot Number	086459		
Description	AMMONIUM NITRATE, A.C.S.		
Country of Origin	Mexico		

Result name	Units	Specifications	Test Value
APPEARANCE		REPORT	COLORLESS TO WHITE GRANULES
ASSAY	%	>= 98.0	99.8
CHLORIDE	ppm	<= 5	<5.0
HEAVY METALS (as Pb)	ppm	<= 5	<5.0
IDENTIFICATION	PASS/FAIL	= PASS TEST	PASS TEST
IGNITION RESIDUE	%	<= 0.01	<0.010
INSOLUBLE MATTER	%	<= 0.005	<0.005
IRON (Fe)	ppm	<= 2	<2.0
NITRITE (NO2)	ppm	<= 5	4.0
PH 5% SOLN @ 25 DEG C		Inclusive Between 4.5 6.0	4.9
PHOSPHATE (PO4)	ppm	<= 5	<5.0
SULFATE (SO4)	%	<= 0.002	<0.0020

Table 3 shows the COA (provided by Fisher Scientific[24]) for the AN used in the Proficiency Test. The results indicate pure AN with very low levels of Fe, nitrite, phosphate, and sulfate.

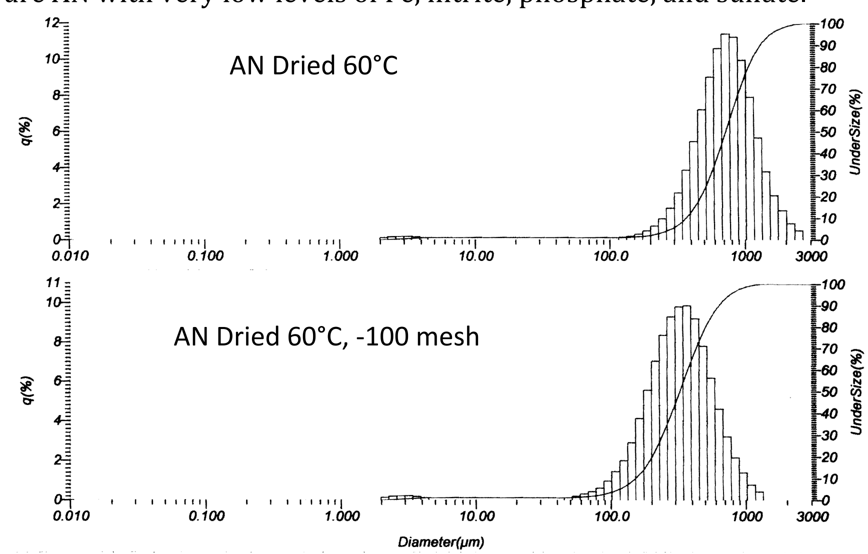

Figure 1. Microtracs laser light scattering particle size distribution for AN (top is AN dried but not size separated, and bottom is AN dried and separated through a 100-mesh sieve).

Figure 1 shows the particle size distribution of the AN after drying and separated and not separated by a 100-mesh sieve[25]. The size distribution is clearly different before the separation, 790 ± 440 μm, compared to after the separation, 360 ± 190 um. The average diameter of the separated fraction is a little larger than expected because the 100-mesh sieve has an opening of 149 μm[26]. Indicating that the aspect ratio of the AN particles is probably very large. In this study, as received is not dried or separated, and dried is dried and separated.

3.2 Impact testing results for AN

Table 4 shows the results of impact testing of AN performed by LANL, LLNL, IHD, and AFRL. Differences in the testing procedures are shown in Table 2, and the notable differences are the sandpaper grit size, amount of sample, and the methods for detection of a positive test. All participants performed data analysis by a modified Bruceton method[27,28]. All participants found AN to be fairly insensitive to impact testing, but the values have a very large range. LLNL used two types of sandpaper—120-grit Si/C wet/dry (LLNL standard) and 180-grit garnet (the IDCA standard). The average values for DH_{50}, in cm, are 156 and 82 for 120-and 180-grit sandpapers, respectively. LANL tested AN both dried and not dried using 180-grit sandpaper and found the material to be completely insensitive to the limit of their equipment. IHD using 180-grit sandpaper found the sensitivity much like what LLNL found for 120-grit sandpaper with an average DH_{50} value of 201 ± 29 cm. AFRL tested a double dried material and found the average DH_{50} value of 60.5 ± 2.5 cm, similar to LLNL results with 180-grit sandpaper.

Table 4. Impact testing results for AN

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120)[5]	1/27/11	23.3	21	157	7.96	0.022
LLNL (120)[5]	1/28/11	23.3	21	155	16.81	0.047
LLNL (180)[5]	2/10/11	22.8	13	76	10.36	0.059
LLNL (180)[5]	2/12/11	22.8	13	88	4.46	0.022
LANL (180)[5]	3/16/11	19.5	< 10	> 320	NA[6]	NA[6]
LANL (180)[5]	3/17/11	19.2	< 10	> 320	NA[6]	NA[6]
LANL (180)[5]	3/18/11	17.5	< 10	> 320	NA[6]	NA[6]
LANL (180)[7]	4/7/11	21.5	18.0	> 320	NA[6]	NA[6]
LANL (180)[7]	4/7/11	21.4	20.0	> 320	NA[6]	NA[6]
LANL (180)[7]	4/7/11	21.3	18.3	> 320	NA[6]	NA[6]
LANL (180)[5]	4/7/11	21.2	24.0	> 320	NA[6]	NA[6]
LANL (180)[5]	4/7/11	21.5	18.1	> 320	NA[6]	NA[6]
LANL (180)[5]	4/7/11	21.5	18.1	> 320	NA[6]	NA[6]
IHD (180)[5]	3/29/11	26	43	178	37.2	0.09
IHD (180)[5]	4/13/11	20	52	192	53.7	0.12
IHD (180)[5]	4/14/11	24	40	233	99.4	0.18
AFRL (180)[5]	5/23/12	22	47	60.5	2.5	0.018
AFRL (180)[5]	5/29/12	22	46	63.0	3.7	0.026
AFRL (180)[5]	5/30/12	22	48	58.1	1.0	0.007

1. Value in parenthesis is grit size of sandpaper (180 is 180-grit garnet dry and 120 is 120-grit Si/Carbide wet); 2. Relative humidity; 3. DH_{50}, in cm, is by a modified Bruceton method, height for 50% probability of reaction; 4. Standard deviation; 5. Dried; 6. Not applicable, outside range of Bruceton Analysis; 7. As received.

Table 5 shows the impact testing of AN performed by LANL and AFRL using the Neyer or D-Optimal method[29]. The LANL data can be divided into two parts, data taken in March 2011 and data taken in April 2011. The data taken in March shows no sensitivity to impact to levels that LANL equipment can test. (Note: the maximum drop heights are the following: LANL, 320 cm; LLNL, 177 cm; IHD, 320 cm; AFRL, 116 cm.) Data taken in April shows slight sensitivity for as received and dried materials. Average DH_{50} values are 304.2 ± 9.2 cm and 304.5 ± 16.7 cm, for as received and dried, respectively. AFRL performed one test that gave a value similar to the average value for DH_{50} determined by the Bruceton method.

Table 5. Impact testing results for AN (Neyer or D-Optimal Method) 180-grit sandpaper

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (180)[5]	3/16/11	19.5	< 10	> 320	NA[6]	NA[6]
LANL (180)[5]	3/17/11	18.9	< 10	> 320	NA[6]	NA[6]
LANL (180)[5]	3/18/11	18.5	< 10	> 320	NA[6]	NA[6]
LANL (180)[7]	4/7/11	22.0	24.5	293.7	8.8	0.013
LANL (180)[7]	4/7/11	21.2	21.0	311.0	26.2	0.037
LANL (180)[7]	4/7/11	21.0	20.8	307.9	12.8	0.018
LANL (180)[5]	4/7/11	21.2	22.3	323.5	67.1	0.091
LANL (180)[5]	4/7/11	21.4	20.9	298.2	10.0	0.015
LANL (180)[5]	4/7/11	21.2	18.1	291.9	23.0	0.014
AFRL (180)[5]	5/23/12	22	47	60.0	13.8	0.102

1. Value in parenthesis is grit size of sandpaper (180 is 180-grit garnet dry); 2. Relative humidity; 3. DH_{50}, in cm, is by the Neyer D-Optimal method, height for 50% probability of reaction; 4. Standard deviation; 5. Dried; 6. Not applicable, outside analysis range; 7. As received.

3.3 Friction testing results for AN

Table 6 shows the BAM Friction testing of AN performed by LANL, LLNL, and IHD. The difference in testing procedures by the three laboratories is shown in Table 2, and the notable differences are in the methods for positive detection. All participants performed data analysis using the threshold initiation level method (TIL)[30]. LANL and LLNL also used a modified Bruceton method[27,28]. All participants found AN to be insensitive to BAM Friction testing.

Table 6. BAM Friction Testing results for AN

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4,5]	s, kg[6]	s, log unit[6]
LLNL[7]	1/27/11	23.3	15	0/10 @ 36	0/10 @ > 36	> 36	NA[8]	NA[8]
LLNL[7]	1/27/11	23.3	15	0/10 @ 36	0/10 @ > 36	> 36	NA[8]	NA[8]
LLNL[7]	1/28/11	23.3	15	0/10 @ 36	0/10 @ > 36	> 36	NA[8]	NA[8]
LANL[7]	3/16/11	19.6	< 10	0/10 @ 36.7	0/10 @ > 36.7	NA[9]	NA[9]	NA[9]
LANL[7]	3/17/11	19.4	< 10	0/10 @ 36.7	0/10 @ > 36.7	NA[9]	NA[9]	NA[9]
LANL[7]	12/08/10	19.4	< 10	0/10 @ 36.7	0/10 @ > 36.7	NA[9]	NA[9]	NA[9]
LANL[7]	3/16/11	19.7	< 10	NA[10]	NA[10]	> 36.7	NA[8]	NA[8]
LANL[7]	3/17/11	19.5	< 10	NA[10]	NA[10]	> 36.7	NA[8]	NA[8]
LANL[7]	3/22/11	19.8	< 10	NA[10]	NA[10]	> 36.7	NA[8]	NA[8]
IHD[7]	4/22/11	22	41	0/10 @ 36.7	0/10 @ > 36.7	NA[11]	NA[11]	NA[11]
IHD[7]	4/22/11	22	41	0/10 @ 36.7	0/10 @ > 36.7	NA[11]	NA[11]	NA[11]
IHD[7]	4/22/11	22	42	0/10 @ 36.7	0/10 @ > 36.7	NA[11]	NA[11]	NA[11]

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. F_{50}, in kg, is by a modified Bruceton method, load for 50% probability of reaction; 5. LLNL uses log spacing and LANL uses liner spacing for the Bruceton up and down method experimentation and data analysis; 6. Standard deviation; 7. Dried; 8. Not applicable, outside range for Bruceton analysis; 9. Not applicable, separate measurements performed for modified Bruceton analysis; 10. Not applicable, separate measurement performed for TIL; 11. Not applicable, Bruceton analysis not performed.

Table 7 shows the ABL Friction testing of AN performed by IHD and AFRL. LANL did not have the system in routine performance at the time. LLNL and SNL do not have ABL Friction testing equipment. IHD and AFRL performed data analysis using the threshold initiation level method (TIL)[30]. IHD also performed a modified Bruceton analysis[27,28]. The data from IHD show that the mixture has some sensitivity, albeit very low. A TIL and one level above TIL are established. The average value for threshold is 0/20 @ 385 psig at 8 fps. In addition, IHD calculated F_{50} values from their data. The average value is

388 ± 16 psig at 8 fps. For the ABL data, IHD was able to establish a TIL unlike for the BAM friction testing. The AFRL data shows a TIL is established in one test case, but not in the others.

Table 7. ABL Friction testing results for AN

Lab	Test Date	T, °C	RH, %[1]	TIL, psig/fps[2,3]	TIL, psig/fps[4]	F_{50}, psig/fps[5]	s, cm[6]	s, log unit[6]
IHD[7]	3/31/11	23	41	0/20 @ 315/8	1/8 @ 420/8	NA[8]	NA[8]	NA[8]
IHD[7]	3/31/11	23	40	0/20 @ 420/8	1/3 @ 560/8	NA[8]	NA[8]	NA[8]
IHD[7]	3/31/11	23	40	0/20 @ 420/8	1/4 @ 560/8	NA[8]	NA[8]	NA[8]
IHD[7]	4/5/11	22	41	NA[9]	NA[9]	406/8	123	0.13
IHD[7]	4/5/11	22	42	NA[9]	NA[9]	376/8	105	0.12
IHD[7]	4/5/11	22	41	NA[9]	NA[9]	383/8	183	0.20
AFRL[7]	5/25/12	22	48	0/20 @ 795/8	2/23 @ 1000/8	NA[10]	NA[10]	NA[10]
AFRL[7]	5/31/12	22	50	0/20 @ 1000/8	NA[11]	NA[10]	NA[10]	NA[10]
AFRL[7]	5/31/12	23	49	0/20 @ 1000/8	NA[11]	NA[10]	NA[10]	NA[10]

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 4. Next level where positive initiation is detected; 5. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% probability of reaction; 6. Standard deviation; 7. Dried; 8. Not applicable, separate measurements done for modified Bruceton analysis; 9. Not applicable, separate measurements performed for TIL analysis; 10. AFRL did not determine a modified Bruceton analysis; 11. AFRL did not measure a TIL.

3.4 Electrostatic discharge testing results for AN

Electrostatic Discharge (ESD) testing of AN was performed by LLNL, LANL, IHD and AFRL. Table 8 shows the results. Differences in the testing procedures are shown in Table 2, and the notable differences are the use of tape and what covers the sample. In addition, LLNL uses a custom built ESD system with a 510-Ω resistor in line to simulate a human body, making a direct comparison of the data from LLNL with data generated by the other participants challenging. All participants performed data analysis using the threshold initiation level method (TIL)[31].

Table 8. Electrostatic discharge testing results for AN

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4,5]	1/26/11	23.3	20	0/10 @ 1.0	0/10 @ 1.0
LLNL[4,5]	1/27/11	23.3	20	0/10 @ 1.0	0/10 @ 1.0
LLNL[4,5]	1/28/11	23.3	16	0/10 @ 1.0	0/10 @ 1.0
LANL[5]	3/16/11	19.6	< 10	0/20 @ 0.125	2/2 @ 0.250
LANL[5]	3/17/11	19.5	< 10	0/20 @ 0.125	1/1 @ 0.250
LANL[5]	3/22/11	18.9	< 10	0/20 @ 0.125	1/1 @ 0.250
IHD[5]	3/28/11	24	42	0/20 @ 0.326	1/3 @ 0.853
IHD[5]	4/7/11	24	43	0/20 @ 0.326	1/1 @ 0.853
IHD[5]	4/11/11	25	54	0/20 @ 0.326	1/2 @ 0.853
AFRL[5]	5/25/12	22	46	0/20 @ 0.38	1/12 @ 0.63
AFRL[5]	6/1/12	22	48	0/20 @ 0.28	1/3 @ 0.31
AFRL[5]	6/1/12	22	48	0/20 @ 0.28	1/4 @ 0.31

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. LLNL used a custom built ESD with a 510-Ω resistor in the discharge unit to mimic the human body. 5. Dried.

Excluding LLNL results, for TIL, AFRL found the material to be the least sensitive based on one measurement. For the average of the measurements, IHD found that AN to be the least sensitive of the participants, while LANL found it to be the most sensitive. The LLNL values using the custom built system show a material with no sensitivity.

3.5 Thermal testing (DSC) results for AN

Differential Scanning Calorimetry (DSC) was performed on the AN by LLNL, LANL, IHD and AFRL. All participating laboratories used different versions of the DSC by TA Instruments. Table 9 shows the data. Results were obtained at a 10°C/min heating rate.

Table 9. Differential Scanning Calorimetry results for AN, 10°C/min heating rate[1,2]

Lab	Test Date	Transition T[1], onset/T_{min} or T_{max}, °C (ΔH, J/g)	Transition T[2], onset/T_{min} or T_{max}, °C (ΔH, J/g)	Transition T[3], onset/T_{min} or T_{max}, °C (ΔH, J/g)	Transition T[4], onset/T_{min} or T_{max}, °C (ΔH, J/g)	Transition T[5], onset/T_{min} or T_{max}, °C (ΔH, J/g)
LLNL[3,4]	1/26/11	52.9/54.1 (-23)		125.9/127.2 (-55)	168.2/168.8 (-73)	238.4/274.5 (-1596)
LLNL[3,4]	1/27/11	53.0/54.4 (-22)		126.0/127.5 (-55)	168.3/168.8 (-75)	231.7/269.5 (-1703)
LLNL[3,4]	1/27/11	52.9/54.5 (-22)		126.0/127.5 (-55)	168.3/168.8 (-75)	254.6/285.9 (-1502)
LLNL[3,5]	1/27/11	51.6/52.8 (-21)	90.5/94.2 (-6)	126.1/127.3 (-55)	168.1/169.2 (-74)	257.6/289.1 (-1378)
LLNL[3,5]	1/28/11	51.7/53.1 (-20)	91.3/92.0 (-3)	126.2/127.7 (-54)	169.0/169.8 (-74)	250.4/283.1 (-1588)
LLNL[3,5]	1/28/11	51.6/53.0 (-20)	91.8/93.4 (-6)	126.2/127.7 (-55)	169.2/169.9 (-74)	258.0/290.8 (-1292)
LLNL[4,6]	1/27/11	53.0/54.3 (-24)		126.1/127.3 (-55)	168.3/168.8 (-74)	235.9/271.4 (-1676)
LLNL[4,6]	1/27/11	53.1/54.2 (-22)		126.0/127.2 (-53)	168.3/168.8 (-72)	229.7/265.5 (-1667)
LLNL[4,6]	1/26/11	53.0/54.2 (-24)		126.0/127.0 (-55)	168.2/168.7 (-70)	231.1/263.4 (-1529)
LLNL[5,6]	1/27/11	51.7/53.0 (-21)	91.8/93.4 (-3)	126.2/127.5 (-55)	169.2/169.9 (-74)	247.2/282.9 (-1559)
LLNL[5,6]	1/28/11	51.7/52.8 (-21)	91.2/94.0 (-5)	126.2/127.6 (-56)	169.2/169.9 (-76)	255.4/286.3 (-1506)
LLNL[5,6]	1/28/11	51.6/53.1 (-19)	91.1/93.8 (-7)	126.2/127.7 (-55)	169.0/169.7 (-75)	244.9/277.0 (-1650)
LANL[3,4]	3/30/11	52.6/53.9 (-24)		126.7/128.7 (-56)	169.0/170.2 (-75)	280.0/310.7 (-535)
LANL[3,4]	3/30/11	52.5/54.1 (-24)		126.8/129.1 (-55)	169.1/170.3 (-75)	281.3/310.9 (-522)
LANL[3,4]	3/30/11	52.5/54.5 (-22)		126.8/128.5 (-53)	168.6/170.5 (-72)	278.6/311.0 (-548)
LANL[3,5]	3/30/11	52.7/54.3 (-21)	90.2/92.0 (-18)	126.6/129.2 (-57)	169.4/169.8 (-79)	281.2/310.2 (-542)
LANL[3,5]	3/30/11	52.7/54.1 (-18)	91.4/92.6 (-17)	126.6/129.0 (-55)	169.0/170.1 (-74)	278.1/311.2 (-539)
LANL[3,5]	3/30/11	53.0/54.5 (-18)	91.0/93.5 (-17)	127.0/129.1 (-54)	169.4/170.7 (-74)	306.0/310.8 (-524)
IHD[3,5]	3/16/11	52.3/53.0 (-3)	92.2/95.1 (-14)	126.1/127.5 (-58)	168.6/169.2 (-77)	287.5/312.4 (-396)
IHD[3,5]	3/16/11	52.1/53.1 (-7)	90.7/94.5 (-9)	126.0/127.2 (-56)	168.7/169.1 (-74)	290.4/325.9 (-466)
IHD[3,5]	3/16/11	52.3/53.0 (-5)	92.6/96.2 (-14)	126.1/127.4 (-60)	168.8/169.1 (-82)	262/325 (-351)
IHD[4,7]	8/6/12	54.4/55.0 (-19)		126.5/128.1 (-40)	166.0/168.3 (-60)	297.4/300.1 (1299)
IHD[4,7]	8/6/12	53.5/54.3 (-16)		126.1/128.2 (-39)	165.8/167.9 (-63)	295.1/298.1 (1430)
IHD[4,7]	8/7/12	54.4/57.5 (-16)		128.1/130.3 (-38)	167.0/169.6 (-56)	293.3/297.0 (1528)
IHD[7,8]	8/7/12	55.8/56.8 (-14)		127.9/131.1 (-40)	164.8/169.1 (-32)	294.6/295.4 (1438)
IHD[7,8]	8/7/12	55.1/56.8 (-20)		127.5/130.3 (-39)	168.2/169.7 (-63)	294.4/296.1 (1510)
IHD[7,8]	8/7/12	54.4/55.2 (-15)		126.9/128.8 (-41)	167.0/169.2 (-60)	293.2/294.3 (1459)
AFRL[3,4]	1/19/11	51.9/53.6 (-21)	85.5/86.6 (-5)	126.6/128.2 (-54)	169.4/170.0 (-73)	292.4/315.9 (-363)
AFRL[3,4]	1/19/11	52.1/53.2 (-24)		126.5/127.8 (-55)	169.4/169.8 (-76)	289.2/315.5 (-397)
AFRL[3,4]	1/20/11	52.2/53.3 (-25)		126.7/128.2 (-56)	169.4/169.7 (-71)	289.7/316.3 (-396)
AFRL[3,5]	6/7/12	47.7/48.5 (-17)	90.4/91.2 (-18)	126.7/129.7 (-57)	169.4/169.8 (-78)	289.6/311.6 (-268)[9]
AFRL[3,5]	6/7/12	46.4/46.6 (-14)	89.5/90.7 (-18)	126.4/128.0 (-56)	169.3/169.6 (-76)	286.3/309.5 (-310)[9]
AFRL[3,5]	6/7/12	47.7/48.6 (-17)	89.4/90.1 (-18)	126.6/129.0 (-57)	169.4/169.9 (-75)	282.2/304.6 (-363)[9]

1. ΔH negative, endothermic; ΔH positive, exothermic; 2. T_{min} = minimum temperature of transition, T_{max} = maximum temperature of transition; 3. Pinhole sample holder; 4. Before drying; 5. after drying; 6. Hermetically sealed sample holder; 7. SWISSI sample holder; 8. Dried 1 month before measurement; 9. Small exothermic transition seen on the high temperature side of this transition.

LLNL used two different sample holders—a standard TA with a 50-μm pinhole lid and the same with a lid without the pinhole that was hermetically sealed. IHD used two different sample holders—a standard TA with 75-μm pinhole lid and a SWISSI high pressure designed to hold 217 bar (3150 psi) at 400°C[31]. LANL and AFRL used the same pinhole sample holder as IHD. Samples were examined as received from manufacturer and dried and separated by IDCA procedures.

Table 9 shows 4 to 5 transitions that have been seen in the literature previously[32-36]. All participants identify these transitions in at least one sample. To appreciate the finer differences in the data based on various parameters, the data in Table 9 was further examined by calculation of the averages, standard deviations, and compilation of maximum and minimum values based on each participant, dried or as received AN, pinhole or sealed sample holder. Figure 2 summarizes the results of these calculations showing the averages and standard deviations of the temperatures (T_{min} or T_{max}) for the Transitions T^1 through T^5 for each of the participants at specific testing conditions in Table 9. The Appendix lists the results of these calculations.

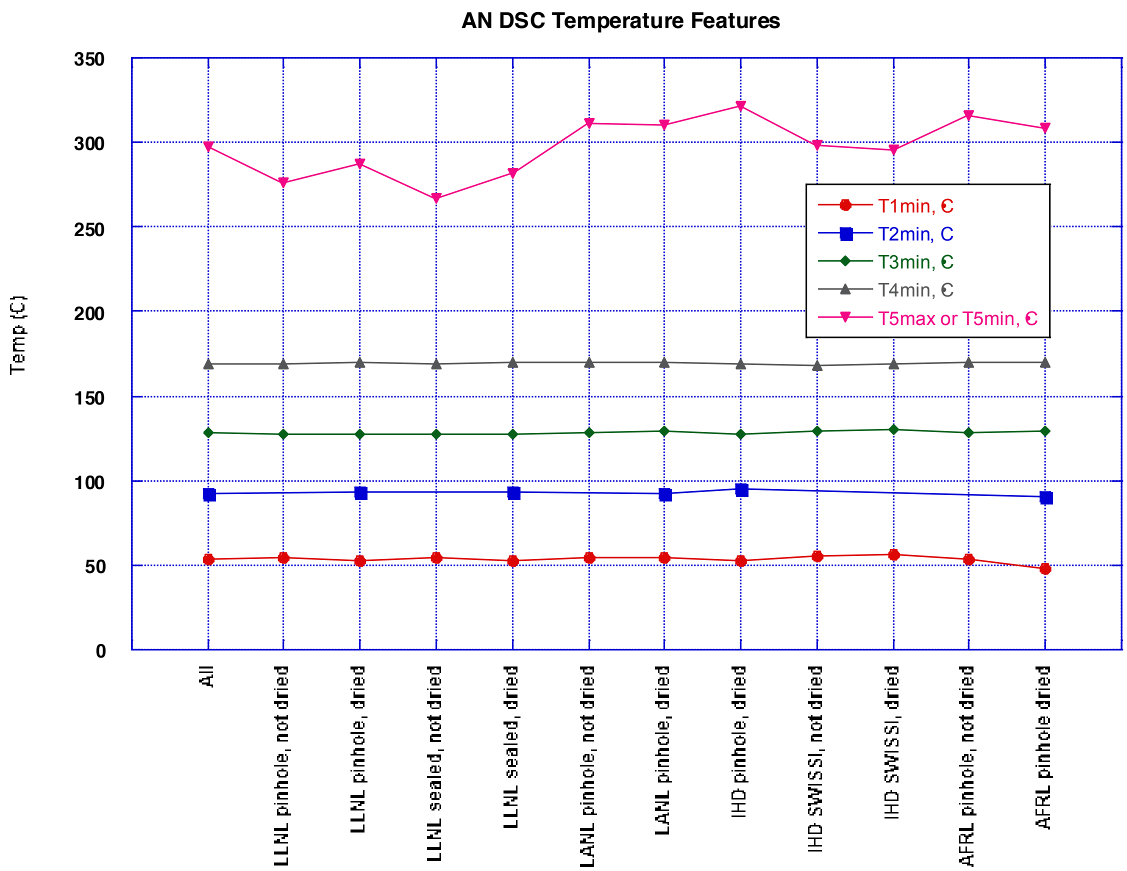

Figure 2. Averages and Standard Deviations for selected groupings of AN DSC Temperature data. The horizontal lines are drawn for convenience of viewing and do not imply any statistical relationship. The x-axis is defines the type of sample holder and the drying status of the AN.

Figure 3 summarizes the results of the calculations showing the averages and standard deviations for the Enthalpies (ΔH_{endo} and ΔH_{exo}) for the Transitions T^1 through T^5 for each of the participants at specific testing conditions in Table 9. The Appendix lists the results of these calculations.

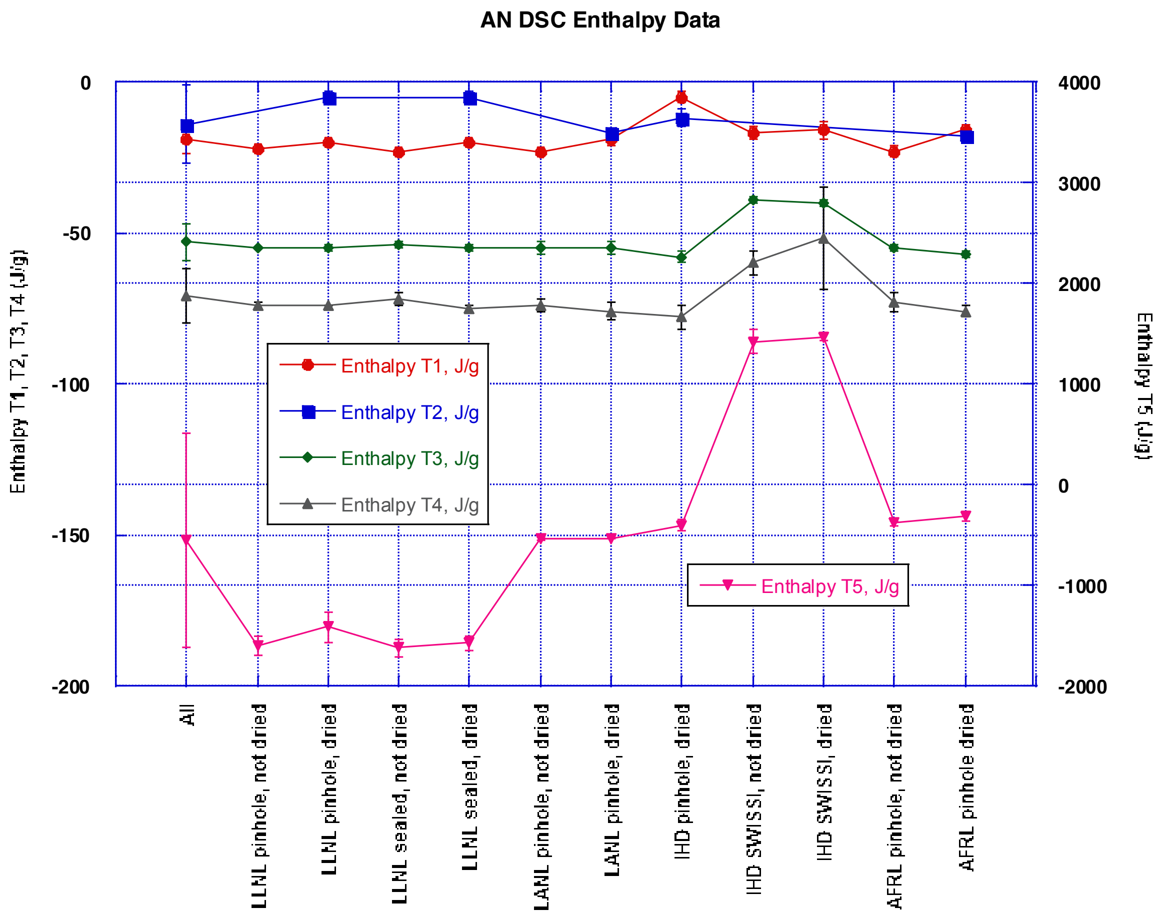

Figure 3. Averages and Standard Deviations for selected groupings of AN DSC Enthalpy data. The horizontal lines are drawn for convenience of viewing and do not imply any statistical relationship. The x-axis is defines the type of sample holder and the drying status of the AN.

To summarize based on transition:

1. Transition T^1 (red circles):
   a. AFRL—onset T and T_{min} for pinhole holder/dried samples are ~ 5 °C lower than the rest of the participants
   b. IHD—enthalpy is less negative for pinhole sample holder/dried sample than the average by about 70%
2. Transition T^2 (blue squares):
   a. LLNL, LANL, IHD, AFRL—onset T, T_{min}, and enthalpy data are present in dried samples only, except for one measurement by AFRL
   b. IHD—onset T, T_{min} and enthalpy data not present in IHD SWISSI sealed sample holder data, dried or not dried sample
   c. LLNL—enthalpy data is 50% less negative than the other participants that measured data for T^2
3. Transition T^3 (green diamonds):
   a. IHD—enthalpy data for SWISSI sealed sample holders/dried and not dried sample about 20% less negative than the enthalpy data from the rest of the participants
4. Transition T^4 (olive green or gray triangles):

a. IHD—enthalpy data for SWISSI sealed sample holders/dried and not dried samples about 20% less negative than the enthalpy data from the rest of the participants

5. Transition T5 (rose inverted triangles):
   a. LLNL, LANL, IHD, AFRL—enthalpy data for pinhole sample holders/dried and not dried samples show endothermic heat flow (LANL, IHD, AFRL ~ -500 J/g, LLNL ~ -1600 J/g)
   b. LLNL—enthalpy data for sealed sample holder/dried and not dried samples show endothermic heat flow at ~ -1600 J/g
   c. IHD—enthalpy data for SWISSI sample holder/dried and not dried samples show exothermic heat flow at ~ 1400 J/g
   d. All—large variations in onset T and T_{min} or T_{max}.

4 DISCUSSION

Table 10 shows the average values for the data for AN from each participant and compares it to corresponding data for standards, RDX Type II Class 5 and PETN done previously. The data for RDX comes from the evaluation of all of the RDX examinations as part of this Proficiency Test[4], and the data for PETN comes from the examination of PETN Class 4 as part of this Proficiency Test[12].

Table 10. Average Comparison Values

	LLNL	LANL	IHD	AFRL
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
AN[2,3]	82[4]	> 320[5]	201[6]	60.5[6]
RDX Type II Class 5[3,7]	22.6	20.9	19.7	15.3
PETN[3,8]	8.3	8.0	9.3	6.8
BAM Friction Testing[9,10]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
AN[11,12]	>36; >36	>36.7; >36.7	>36.7; >36.7	ND[13]; ND[13]
RDX Type II Class 5[7]	16.3; 23.4	14.8; 18.3	15.1; 19.3	ND[13]; ND[13]
PETN[8]	6.4; 10.5	4.9, 8.5	4.3, 6.9	ND[13]; ND[13]
ABL Friction Testing[14-17]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig
AN[18]	ND[13]; ND[13]	ND[13]; ND[13]	385[19]; 388[19]	> 798[19]; ND[13]
RDX Type II Class 5[7]	ND[13]; ND[13]	ND[13]; ND[13]	80; 179	93; ND[13]
PETN[8]	ND[13]; ND[13]	ND[13]; ND[13]	7.7, 42	ND[13]; ND[13]
Electrostatic Discharge[20]	TIL, Joules	TIL, Joules	TIL, Joules	
AN[21,22]	0/10 @ 1.0[23]	0/20 @ 0.125[24]	0/20 @ 0.326[24]	0/20 @ 0.313[24]
RDX Type II Class 5[7]	0/10 @ 0.038[24]	0/20 @ 0.027[24]	0/20 @ 0.066[24]	0/20 @ 0.044[24]
PETN[8]	0/10 @ 0.033[24]	0/20 @ 0.025[24]	0/20 @ 0.219[24]	0/20 @ 0.076[24]

1. DH_{50}, in cm, is by a modified Bruceton method, height for 50% probability of reaction; 2. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.8; 13), LANL (17.5-21.5; <10-24.0), IHD (20-26; 40-52), AFRL (22; 46-48); 3. 180-grit sandpaper; 4. Average of 2 data points from Table 4; 5. Average of 9 data points from Table 4; 6. Average of 3 data points from Table 4; 7. From reference 4; 8. From reference 12; 9. Threshold Initiation Level (TIL) is the weight (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 10. F_{50}, in kg, is by a modified Bruceton method, weight for 50% probability of reaction; 11. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.3; 15), LANL (19.4-19.8; <10), IHD (22; 41-42); 12. Average of measurements from Table 6; 13. ND = Not determined; 14. LLNL and LANL did not perform measurements; 15. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 16. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% probability of reaction; 17. Measurements performed at 8 fps; 18. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (22-23; 40-42), AFRL (22-23; 48-50); 19. Average of 3 measurements in Table 7; 20. Threshold Initiation Level (TIL) is the energy (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 21. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.3; 16-20), LANL (18.9-19.6; <10, IHD (24-25; 42-54), AFRL (22; 46-48); 22. Average of 3 measurements from Table 8; 23. LLNL has 510-Ω resistor in circuit; 24. ABL ESD apparatus.

4.1 Comparison of participating laboratory testing results for AN

Impact sensitivity. All the impact data in Table 10 for AN was taken using 180-grit garnet sandpaper. (LLNL also used 120-grit Si/C sandpaper and these results are listed in Table 3, but not summarized in Table 10.) All participants found AN insensitive, but in widely differing degrees. LANL found the AN to have no sensitivity (up to 320 cm drop height), while LLNL, IHD, and AFRL were able to measure some sensitivity using the Bruceton method. The data taken with the 120-grit Si/C wet sandpaper by LLNL also shows AN also to be insensitive, but having a much different DH_{50}. This has been seen before in the IDCA proficiency Test where the 120-grit sandpaper used by LLNL indicates a substantially less sensitive material than the same testing with 180-grit sandpaper[5-8,10,11,37]. The reasons for the wide range in sensitivity results are postulated below. Also, by LANL data from the Neyer analysis, drying of AN has no effect on the impact sensitivity.

Friction sensitivity. For BAM Friction, LLNL, LANL, and IHD found AN to be insensitive by both TIL and the modified Bruceton method (beyond the testing capabilities of the BAM equipment). For ABL Friction, IHD and AFRL found the AN to be insensitive, although IHD was able to establish a TIL and calculate a F_{50} value.

ESD. LANL, IHD and AFRL have similar ABL ESD systems that differ by vintage. This difference is reflected through the ability to set stimulation levels. Interestingly, AFRL and IHD testing show similar spark sensitivity results for AN and they have the newest and the oldest equipment. LANL shows a spark sensitivity of about one half the value. LLNL is not compared in this group because, for this testing, LLNL used a custom built system that has a 510-Ω resistor in the circuit, making the direct comparison with other participants difficult.

Thermal sensitivity. All participants found AN to have either four or five thermal transitions. Although there are some differences in position and intensity of the transitions, most measurements agree independent of the testing condition differences. However, there are some differences and these will be discussed below.

4.2 Comparison of average testing values for AN with standards

Table 10 shows the comparison of the impact, friction and ESD sensitivity of AN with the standards RDX Type II Class 5 and PETN Class 4.

Impact sensitivity. All participants found AN to be much less sensitive than the RDX and PETN. LANL did find AN to have some sensitivity when using the Neyer method, but this sensitivity was still near the testing limit of the drop hammer. AFRL found the sensitivity by the Neyer method to be about the same as with the modified Bruceton method.

Friction sensitivity. LLNL, LANL and IHD found AN to be insensitive when testing with BAM friction, while the standards were found to have some sensitivity. IHD found AN to have some sensitivity when using the ABL system, but this sensitivity was much less than the RDX and PETN tested under the same conditions. AFRL found virtually no sensitivity of AN when using the ABL system.

Spark sensitivity. All participants found AN to be less sensitive than the RDX Type II Class 5 and PETN Class 4 standards. LANL, IHD and AFRL did measure some spark sensitivity for the AN, but at a level several times less than the standards. LLNL measured no sensitivity on the custom system with a 510-Ω resistor in the circuit.

Thermal sensitivity. The majority of the DSC measurements on AN indicate only endothermic transitions. If this is accurate behavior, then AN is insensitive compared to the standards. However, in the high-pressure sample holder, IHD found the highest temperature transition, T^5, to be exothermic, with a T_{max} around 300° C and ΔH_{exo} near 1400 J/g. If this is accurate behavior, then AN is thermally sensitive but still less sensitive than either standard. The T_{max} and ΔH_{exo} for RDX and PETN, respectively are: ~ 240°C, ~ 2200 J/g; ~ 205 °C, ~ 1100 J/g[4,12].

4.3 Drop hammer reality when testing AN

Table 4 shows a wide variety of results for the impact testing of AN. The results seem to be participant dependent. Even though all participants have similar equipment (not identical), the results are still quite varied.

RDX has been studied by the IDCA multiple times in the Proficiency Test[2-4]. From these tests, there has been sufficient data collected to perform statistical analyses[38]. For RDX, the order of drop hammer sensitivity was found to be LLNL < LANL < IHD < AFRL. However, some analyses indicate that LLNL and LANL results are statistically the same and that AFRL is statistically different (more sensitive) and IHD bridges in between[38]. In this study on AN, although not evaluated statistically, the order for drop hammer sensitivity was found to be LANL<< IHD << LLNL < AFRL, where the magnitudes of the differences are much more than in the RDX case. Temperature and humidity are probably not the reason because the orders do not follow either parameter. For temperature, the order is LANL > AFRL > LLNL > IHD; for humidity the order is LANL < LLNL < IHD = AFRL.

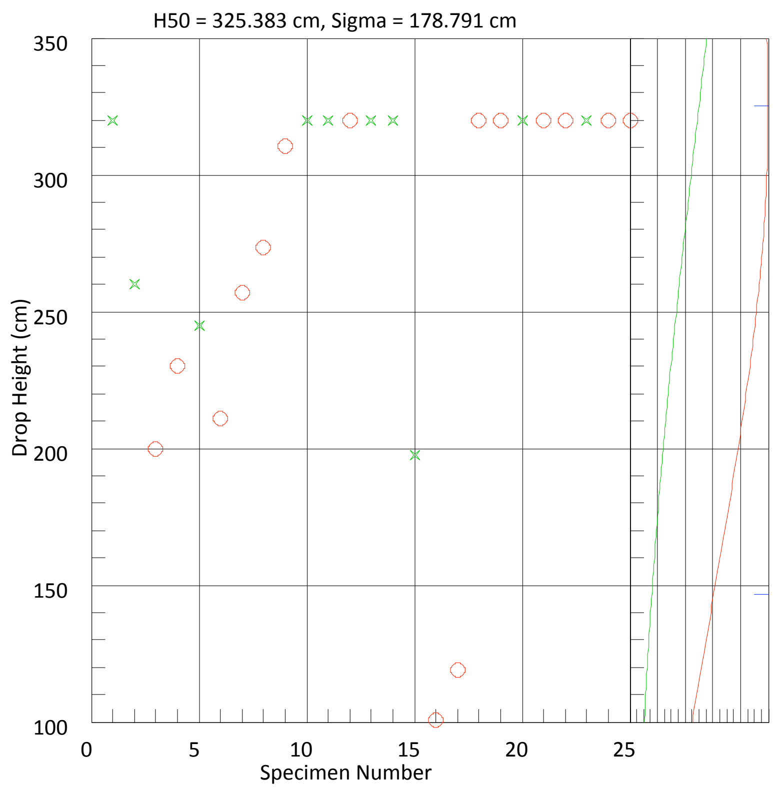

Figure 4. Testing of AN by Neyer method (x = positive event, o = negative event)

A potential source for these differences in results can be found in the positive/negative reaction ("go-no/go") determination process. AN appears to be a very difficult material to test in the drop hammer experiment. The problem seems to be with the determination of a positive/negative event based on sound. At large drop heights, materials that yield little noise when reacting are difficult to discern because the background noise is so great. Figure 4 shows the positive/negative determination graph for a Neyer determination on AN. 25 trials were performed on the AN (180-grit garnet sandpaper, 35 mg samples). The detection method used was a sound threshold over background. A green x marks a positive reaction and a red o marks a negative reaction. Clearly the range of positive and negative events is very large. There are positive events as low as 200 cm and negative events as high as 320 cm (upper limit of the equipment). This material has very low sensitivity and as a result, the background from the high drop height can interfere with determining a positive and negative event. For the sound meter, the drop hammer produces 110 dB on the average for a negative reaction and 122 dB on the average for a positive reaction. To illustrate the issue further, Figure 5 shows photographs of two tests that were considered a positive event. The photographs were taken in the dark with the aperture wide open (f1.8) and with maximum speed (6400). The photograph on the left shows an event selected as positive by sound. This clearly shows light of reaction. However, the photograph on the right side was also identified by sound as a positive event, but no light was visible. Visibly, it would be considered a negative or no-go, but was recorded by sound as a positive event. Many of the events that were considered positive did not exhibit light. Higher order was verified in some cases by inspection of the sandpaper after the test.

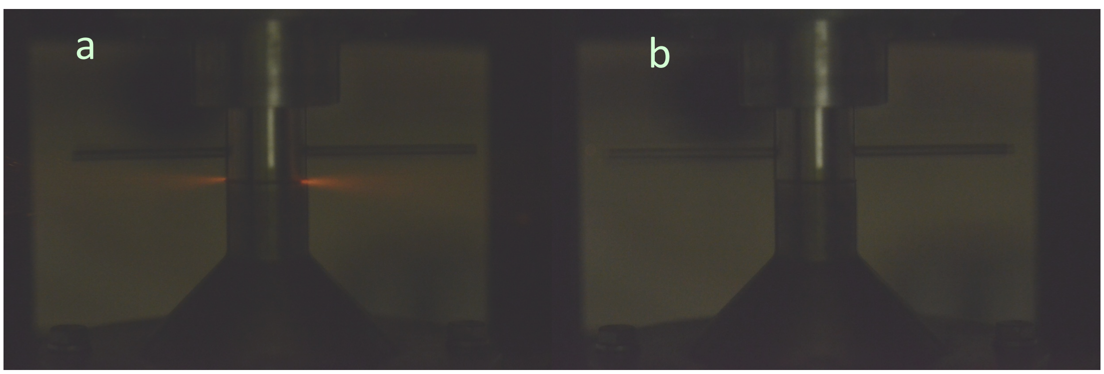

Figure 5. Photographs of positive events in the drop hammer testing of AN, Neyer method. Both were considered a positive event by sound. The left shows visible evidence, the right does not.

To complicate matters among participants even further, is that positive/negative determination is not uniformly conducted. LANL uses a sound detection system with a microphone that is a meter away. Assessment of positive or negative can be overridden by other input, such as personal decisions by the operator and/or by photography. LLNL uses a different type of microphone that is 6 inches from the anvil. Assessment of a positive or negative event can be overridden by personal decisions by the operator. IHD and AFRL use operator only decision-making.

Issues such as these can account for the wide differences in results in Table 4. They also highlight the need for a standard method of detection in the testing community.

4.4 Thermal behavior of AN

Transition T^5—endothermic or exothermic. The participants had varied results for the thermal decomposition of AN. For the high temperature transition T^5, the temperature ranges and enthalpy values were different (LANL and IHD enthalpy values were about 1/3 of the LLNL values). There was also a disagreement between DSC behavior by observation and intuition because the region where the oxidizer decomposes, T^5, was exhibiting endothermic decomposition where exothermic decomposition is expected. Only the IHD data with the SWISSI high-pressure sample holders exhibited exothermic response. As well, Figure 6 shows the same controversy from the literature. The left profile is from Gunawan and Zhang[36] and the right profile is from Oxley et al.[34]. The profiles are similar except for exact minimum temperature of the endothermic features and the high temperature transition is an endothermic feature in Gunawan and Zhang and is an exothermic feature in Oxley et al. The former issue can be explained by the different heating rates. An exothermic feature is expected for the latter issue because the feature is due to an energetic material decomposing.

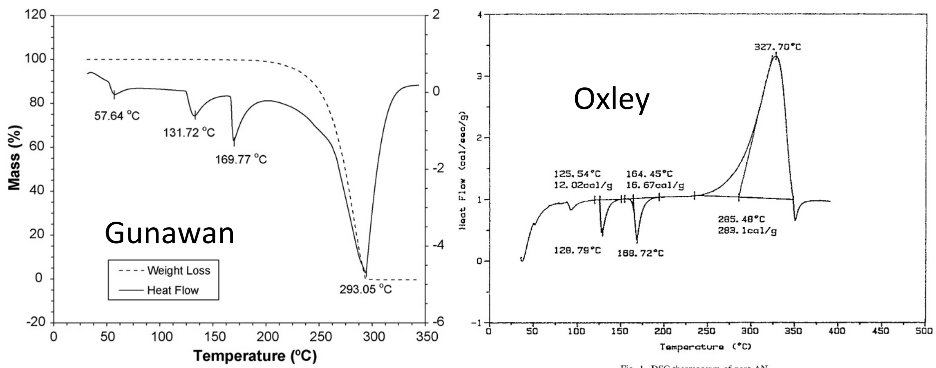

Figure 6. DSC profiles of AN by Gunawan and Zhang (2009)[36] and by Oxley et al. (2002)[34].

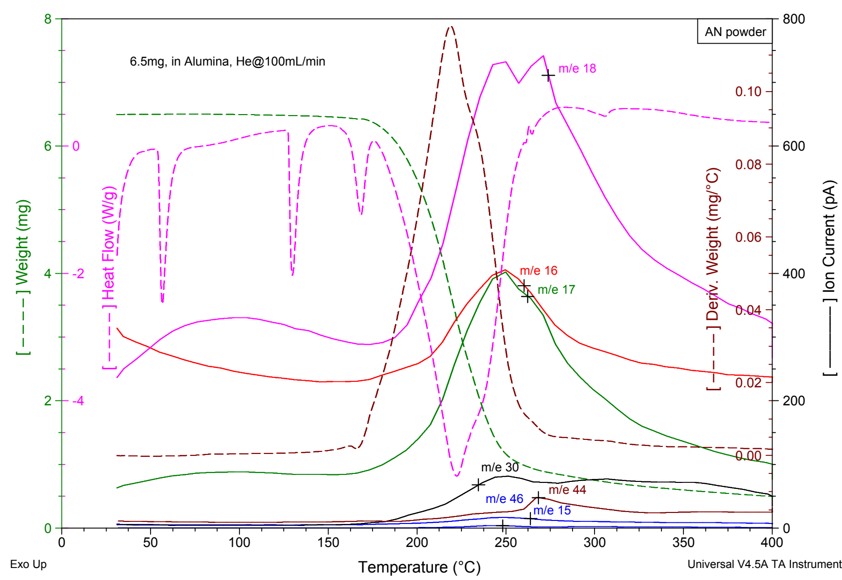

Figure 7. MS of volatile gases during the heating of AN at 10°C/min

To resolve this apparent conflict with IDCA data, AN was heated at 10°C/min and the volatile emissions were monitored by MS. Figure 7 shows the results. AN decomposes at T^5 (around 240°C), and producing volatile gases. The light masses (m/e 15, 16, 17, 18, 30, 44, and 46) were monitored and are observed as decomposition products of the ammonium ion and the nitrate ion (H_2O, NH_3, NO, N_2O, NO_2). Note also the sample looses about 85 % of weight during the T^5 transition.

These differences in T^5 among the participants are simply explained by the type of DSC sample holder that is used for the measurement. The pinhole vented sample holders allow for the gases to escape causing evaporative cooling, an endothermic event, which overrides any positive heat flow from decomposition, an exothermic event. When the gases are not allowed to escape, an exothermic feature is observed instead because of net exothermic heat flow.

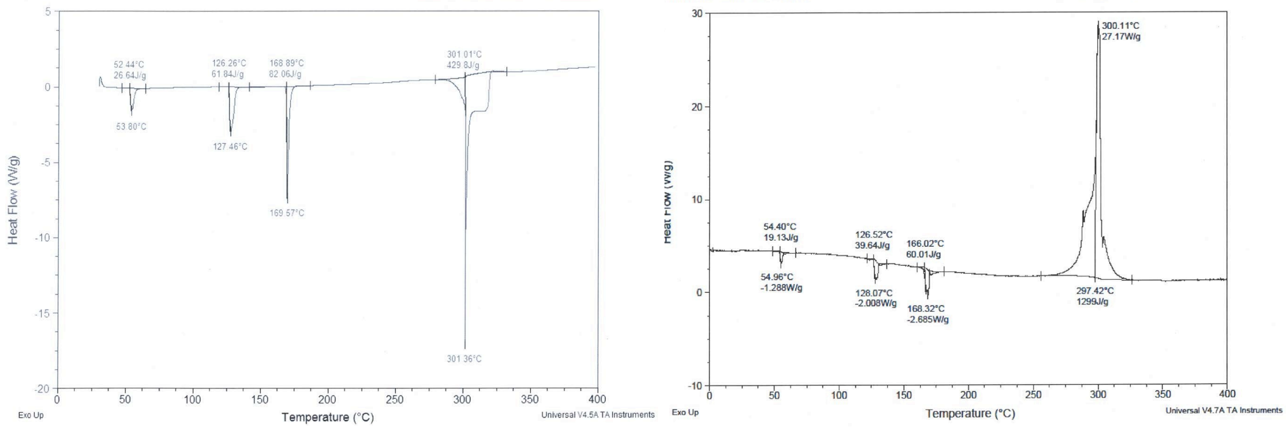

Figure 8. DSC of AN using a pinhole sample holder (left) and SWISSI sealed holder (right) at 10°C/min heating rate.

The left side of Figure 8 shows AN sample heated in the standard Proficiency Test DSC sample holder with a pinhole sample lid. The right side of Figure 8 shows the AN sample heated in a gold sealed sample holder (Gold High Pressure, SWISSI crucibles sold in US by Fauske[31]). The high temperature exothermic feature is clearly seen.

It should be noted that LLNL also made measurements with a sealed sample holder. This sample holder uses the standard TA body but instead of the laser-drilled pinhole in the lid, it uses a non-vented lid. Based on the results above, it is speculated that even though this sample holder is hermetically sealed (as described by the manufacturer), it is really only pressure rated to a few pounds and that it ruptures before or at the onset of T^5. The end result is an endothermic response for T^5.

Drying of AN. From the initial planning of the Proficiency Test, a critical parameter was that each participating laboratory use identical test materials and preparation. This way, material variability could be reduced or eliminated. AN absorbs some moisture[39]. The humidity varies significantly among the IDCA laboratories and is not rigorously controlled. Moisture also affects the crystalline and reactive properties of AN. A drying and storage procedure is probably the best way to control this moisture effect, so a drying procedure was developed[40] that would be effective and could be safely accommodated by all the participants (for example, safety approval to heat above 80°C is a very time consuming adventure at the DOE National Laboratories).

AN has a very complicated thermal sensitivity. Table 11 shows the temperature phase transitions that occur in a heating cycle. AN becomes very sensitive towards shock in the presence of water (humidity) when it is subjected to temperature cycling through the 32°C phase change. KNO_3 (a phase stabilizer) and desiccants, such $Mg(NO_3)_2$ and $Al_2(SO_4)_3$ are added to AN to stabilize it against this phase change.

Table 11. Selected physical conformations of AN[41,42]

System	Temperature Range	State	Volume change
-	> 169.6	Liquid	-
I	169.2 to 125.2	Cubic	+ 2.1
II	125.5 to 84.2	Tetragonal	-1.3
III	84.2 to 32.3	a-rhombic	+3.6
IV	32.3 to -16.8	b-rhombic	-2.9
V	-16.8	tetragonal	-

A large volume change between the phases of IV→III creates a lot of pores upon thermal cycling. These heterogeneities are the source for the increased sensitivity (hot spots). As the water content of AN is reduced below 0.2%, the IV→III phase transition temperature increases and the III→IV phase transition decreases; below 0.01% water, IV does not form III at all resulting in a direct II →IV transition[41,42].

Previous studied materials in the IDCA were subjected to the following drying conditions[14]—16 hours at 60°C then cooling and storing in a desiccator. This process had been easily implemented by the IDCA participants, so it seemed attractive to apply to AN. However, given the complexity of the phase relationship, a sample AN was put through several thermal cycles to see if drying would have deleterious effect[43].

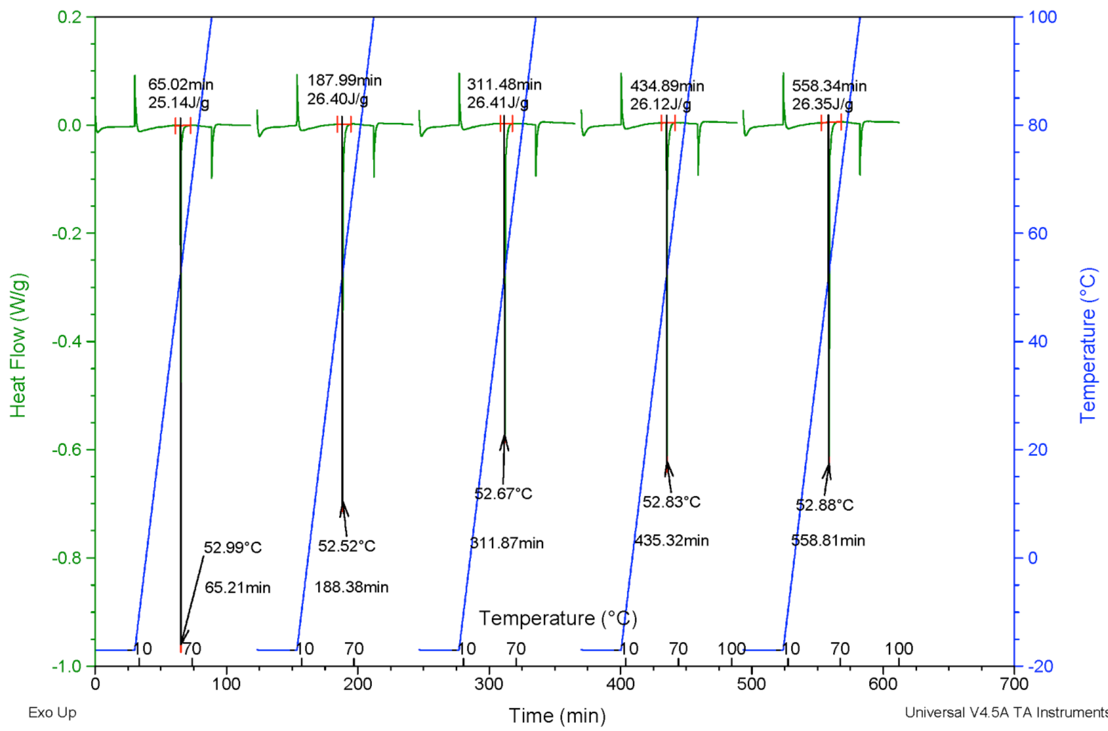

Figure 9. DSC of AN cycled several times through -10 to 100°C.

AN 1.6 g sample of dried AN was subjected to heating at 10°C/min from RT to 100°C then cooled to room temperature in a TA2000 DSC. The temperature and heat flow were monitored in the ascending heating. Figure 9 shows the results.

For the all the heating cycles, transition T^1 and T^2 (a described in Table 9) are clearly visible. Only the features of T^1 are recorded on the figure. For the five cycles, the average T^1 specifications are 52.8 ± 0.2 °C and 26.1 ± 0.5 J/g. These values show that repeated heating to 100°C do not seem to affect the overall stability of the AN, indicating the standard IDCA method would adequate for drying the AN material.

As a further test, AN was heated to 120°C, using a 5°C/min heating rate for 1 h followed by holding the sample at a 120°C for another 2 h. The AN as received from the manufacturer lost 7.7% of the mass[44]. When dried at 90°C for 20 h, the AN as received from the manufacturer looked unchanged. DSC was performed on both materials, and the transitions of relevance before and after drying to 90°C are: before drying T^1—52.8, °C, -25.5 J/g; T^3—126.8 °C, -57.4 J/g; T^4—169.4 °C, -79.3 J/g; after drying T^1—52.5 °C, -23.7 J/g; T^3—126.9 °C, -52.9 J/g; T^4—169.6 °C, -74.6 J/g. The temperature and enthalpy values of the before drying sample and the corresponding values after drying sample are essentially the same. Note also the absence of transition T^2 in both cases, the same as the as not dried material in Table 9.

5 CONCLUSIONS

Conclusions from this study are:

1. Impact testing of AN
    a. Each participant found AN to be much less sensitive than the RDX standard
    b. The DH_{50} values varied significantly (although still insensitive) among participants
    c. Testing of AN is problematic for determining a positive event
    d. LANL found drying has no affect on the impact sensitivity (Neyer method)
2. Friction testing of AN
    a. LLNL, LANL and IHD found AN completely insensitive with BAM friction
    b. AFRL found AN completely insensitive with ABL friction
    c. IHD found AN much less sensitive that the RDX standard when using ABL friction
3. Spark testing
    a. LANL, IHD, and AFRL found AN much less sensitive than the RDX and PETN standards
    b. LLNL found AN insensitive
4. Thermal testing of AN
    a. Each participant found the AN to be insensitive to thermal excursions when using vented sample holders
    b. LLNL found AN to be insensitive to thermal excursions when using a sealed sample holder
    c. IHD found AN to be exothermic around 300°C when using a high pressure sealed sample holder
    d. IHD found AN to be thermally less sensitive to RDX and PETN.

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ABREVIATIONS, ACRONYMS AND INITIALISMS

-100	Solid separated through a 100-mesh sieve
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AR	As received (separated through a 40-mesh sieve)
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
DH_{50}	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
ESD	Electrostatic Discharge
F_{50}	The weight or pressure used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
fps	feet per second
H	Chemical symbol for hydrogen
H_2O	Chemical formulation for water

HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
j	joules
$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
kg	kilograms
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
N	Chemical symbol for nitrogen
$NaClO_3$	Sodium Chlorate
NSWC	Naval Surface Warfare Center
O	Chemical symbol for oxygen
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RH	Relative humidity
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
s	Standard Deviation
SEM	Scanning Electron Micrograph
Si	silicon
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event

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Appendix

Table A.1. Temperature and Enthalpy Averages and Ranges of DSC data Transition T^1 for AN

Participant and Parameters	Onset T^1, °C	T^1_{min}, °C	Enthalpy T^1, J/g
All	52.3 ± 1.9 46.4 to 55.8	53.6 ± 2.2 46.6 to 57.5	-19 ± 5 -3 to -25
LLNL pinhole, not dried	52.9 ± 0.1 52.9 to 53	54.3 ± 0.2 54.1 to 54.5	-22 ± 1 -23 to -22
LLNL pinhole, dried	51.6 ± 0.1 51.6 to 51.7	53.0 ± 0.2 52.8 to 53.1	-20 ± 1 -21 to -20
LLNL sealed, not dried	53.0 ± 0.1 53.0 to 53.1	54.2 ± 0.1 54.2 to 54.3	-23 ± 1 -24 to -22
LLNL sealed, dried	51.7 ± 0.1 51.6 to 51.7	53.0 ± 0.2 52.8 to 53.1	-20 ± 1 -21 to -19
LANL pinhole, not dried	52.5 ± 0.1 52.5 to 52.6	54.2 ± 0.3 53.9 to 54.5	-23 ± 1 -24 to -22
LANL pinhole, dried	52.8 ± 0.2 52.7 to 53.0	54.3 ± 0.2 54.1 to 54.5	-19 ± 2 -21 to -18
IHD pinhole, dried	52.2 ± 0.1 52.1 to 52.3	53.0 ± 0.1 52.1 to 52.3	-5 ± 2 -7 to -3
IHD SWISSI, not dried	54.1 ± 0.5 53.5 to 54.4	55.6 ± 1.7 54.3 to 57.5	-17 ± 2 -19 to -16
IHD SWISSI, dried	55.1 ± 0.7 54.4 to 55.8	56.2 ± 0.9 55.2 to 56.8	-16 ± 3 -20 to -14
AFRL pinhole, not dried	52.1 ± 0.2 51.9 to 52.2	53.4 ± 0.2 53.2 to 53.6	-23 ± 2 -25 to -21
AFRL pinhole dried	47.3 ± 0.8 46.4 to 47.7	47.9 ± 1.1 46.6 to 48.6	-16 ± 2 -17 to -14

Table A.2. Temperature and Enthalpy Averages and Ranges of DSC data Transition T^2 for AN

Participant and Parameters	Onset T^2, °C	T^2_{min}, °C	Enthalpy T^2, J/g
All	90.7 ± 1.6 85.5 to 92.6	92.7 ± 2.3 86.6 to 96.2	-14 ± 13 -57 to -3
LLNL pinhole dried	91.2 ± 0.7 90.5 to 91.8	93.2 ± 1.1 92.0 to 94.2	-5 ± 2 -6 to -3
LLNL sealed dried	91.4 ± 0.4 91.1 to 91.8	93.7 ± 0.3 93.4 to 94	-5 ± 2 -7 to -3
LANL pinhole dried	90.9 ± 0.5 90.2 to 91.4	92.7 ± 0.8 92.0 to 93.5	-17 ± 1 -18 to -17
IHD pinhole dried	91.9 ± 1.0 90.7 to 92.6	95.3 ± 0.9 94.5 to 96.2	-12 ± 3 -14 to -9
AFRL pinhole dried	89.8 ± 0.6 89.4 to 90.4	90.7 ± 0.6 90.1 to 91.2	-18 ± 0 -18 to -18

Table A.3. Temperature and Enthalpy Averages and Ranges of DSC data Transition T^3 for AN

Participants and Parameters	Onset T^3, °C	T^3_{min}, °C	Enthalpy T^3, J/g
All	126.5 ± 0.5 125.9 to 128.1	128.3 ± 1.0 127.0 to 131.1	-53 ± 6 -60 to -38
LLNL pinhole not dried	126.0 ± 0.1 125.9 to 126.0	127.4 ± 0.2 127.2 to 127.5	-55 ± 0 -55
LLNL pinhole dried	126.2 ± 0.1 126.1 to 126.2	127.6 ± 0.2 127.3 to 127.7	-55 ± 1 -55 to -54
LLNL sealed not dried	126.0 ± 0.1 126.0 to 126.1	127.2 ± 0.2 127 to 127.3	-54 ± 1 -55 to -53
LLNL sealed dried	126.2 ± 0.0 126.2 to 126.2	127.6 ± 0.1 127.5 to 127.7	-55 ± 1 -56 to -55
LANL pinhole not dried	126.8 ± 0.1 126.7 to 126.8	128.8 ± 0.3 128.5 to 129.1	-55 ± 2 -56 to -53
LANL pinhole dried	126.7 ± 0.2 126.6 to 127	129.1 ± 0.1 129 to 129.2	-55 ± 2 -57 to -54
IHD pinhole dried	126.1 ± 0.1 126.0 to 126.1	127.3 ± 0.2 127.2 to 127.5	-58 ± 2 -60 to -56
IHD SWISSI not dried	126.9 ± 1.1 126.1 to 128.1	128.9 ± 1.2 128.1 to 130.3	-39 ± 1 -38 to -40
IHD SWISSI dried	127.4 ± 0.5 126.9 to 127.9	130.1 ± 1.2 128.8 to 131.1	-40 ± 1 -39 to -41
AFRL pinhole not dried	126.6 ± 0.1 126.5 to 126.7	128.1 ± 0.2 127.8 to 128.2	-55 ± 1 -54 to -56
AFRL pinhole dried	126.5 ± 0.2 126.4 to 126.7	128.9 ± 0.9 128.0 to 129.7	-57 ± 1 -57 to -56

Table A.4. Temperature and Enthalpy Averages and Ranges of DSC data Transition T^4 for AN

Participant and Parameter	Onset T^4, °C	T^4_{min}, °C	Enthalpy T^4, J/g
All	168.5 ± 1.1 164.8 to 169.4	169.5 ± 0.6 167.9 to 170.7	-71 ± 9 -82 to -32
LLNL pinhole not dried	168.3 ± 0.1 168.2 to 168.3	168.8 ± 0.0 168.8 to 168.8	-74 ± 1 -75 to -73
LLNL pinhole dried	168.8 ± 0.6 168.1 to 169.9	169.6 ± 0.4 169.2 to 169.9	-74 ± 0 -74 to -74
LLNL sealed not dried	168.3 ± 0.1 168.2 to 168.3	168.8 ± 0.1 168.7 to 168.8	-72 ± 2 -74 to -70
LLNL sealed dried	169.1 ± 0.1 169 to 169.2	169.8 ± 0.1 169.7 to 169.9	-75 ± 1 -76 to -74
LANL pinhole not dried	168.9 ± 0.3 168.8 to 169.1	170.3 ± 0.2 170.2 to 170.5	-74 ± 2 -75 to -72
LANL pinhole dried	169.3 ± 0.2 169.0 to 169.4	170.2 ± 0.5 169.8 to 170.7	-76 ± 3 -79 to -74
IHD pinhole dried	168.7 ± 0.1 168.6 to 168.8	169.1 ± 0.1 169.1 to 169.2	-78 ± 4 -82 to -74
IHD SWISSI not dried	166.3 ± 0.6 165.8 to 167.0	168.6 ± 0.9 167.9 to 169.6	-60 ± 4 -63 to -56
IHD SWISSI dried	166.7 ± 1.7 164.8 to 168.2	169.3 ± 0.3 169.1 to 169.7	-52 ± 17 -63 to -32
AFRL pinhole not dried	169.4 ± 0.0 169.4 to 169.4	169.8 ± 0.2 169.7 to 170	-73 ± 3 -76 to -71

AFRL pinhole dried	169.4 ± 0.1 169.3 to 169.4	169.8 ± 0.2 169.6 to 169.9	-76 ± 2 -78 to -75

Table A.5. Temperature and Enthalpy Averages and Ranges of DSC data Transition T^5 for AN

Participants and Parameters	Onset T^5, °C	T^5_{max} or T^5_{min}, °C	Enthalpy T^5, J/g
All	272.0 ± 23.2 229.7 to 306	297.6 ± 17.4 263.4 to 325.9	-550 ± 1066 -1703 to 1528
LLNL pinhole not dried	241.6 ± 11.8 231.7 to 254.6	276.6 ± 8.4 269.5 to 285.9	-1600 ± 101 -1703 to -1502
LLNL pinhole dried	255.3 ± 4.3 250.4 to 258	287.7 ± 4.0 283.1 to 290.8	-1419 ± 152 -1588 to -1292
LLNL sealed not dried	232.2 ± 3.3 229.7 to 235.9	266.8 ± 4.1 263.4 to 271.4	-1624 ± 82 -1676 to -1529
LLNL sealed dried	249.2 ± 5.5 244.9 to 255.4	282.1 ± 4.7 277 to 286.3	-1572 ± 73 -1650 to -1506
LANL pinhole not dried	280.0 ± 1.4 278.6 to 281.3	310.9 ± 0.2 310.7 to 311	-535 ± 13 -548 to -522
LANL pinhole dried	288.4 ± 15.3 278.1 to 306	310.7 ± 0.5 310.2 to 311.2	-535 ± 10 -542 to -524
IHD pinhole dried	280.0 ± 15.7 262.0 to 290.4	321.1 ± 7.5 312.4 to 325.9	-404 ± 58 -466 to -351
IHD SWISSI not dried	295.3 ± 2.1 293.3 to 297.4	298.4 ± 1.6 297 to 300.1	1419 ± 115 1299 to 1528
IHD SWISSI dried	294.1 ± 0.8 293.2 to 294.6	295.3 ± 0.9 294.3 to 296.1	1469 ± 37 1438 to 1510
AFRL pinhole not dried	290.4 ± 1.7 289.2 to 292.4	315.9 ± 0.4 315.5 to 316.3	-385 ± 19 -397 to -363
AFRL pinhole dried	286.0 ± 3.7 282.2 to 289.6	308.6 ± 4.0 304.6 to 311.6	-314 ± 48 -363 to -268

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